(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) Veröffentlichungsnummer:
(11) Publication number: **EP 1 135 227 A0**
(11) Numéro de publication:

Internationale Anmeldung veröffentlicht durch die Weltorganisation für geistiges Eigentum unter der Nummer:

WO 00/30786 (art. 158 des EPÜ).

International application published by the World Intellectual Property Organisation under number:

WO 00/30786 (art. 158 of the EPC).

Demande internationale publiée par l'Organisation Mondiale de la Propriété sous le numéro:

WO 00/30786 (art. 158 de la CBE).

PCT WORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 7 : B22D 41/18 — A1

(11) International Publication Number: WO 00/30786

(43) International Publication Date: 2 June 2000 (02.06.00)

(21) International Application Number: PCT/BE99/00146

(22) International Filing Date: 16 November 1999 (16.11.99)

(30) Priority Data:

9800837	20 November 1998 (20.11.98)	BE
9800838	20 November 1998 (20.11.98)	BE

(71) Applicant *(for all designated States except US)*: VESUVIUS CRUCIBLE COMPANY [US/US]; Suite 200, Foulk Road 103, Wilmington, DE 19803 (US).

(72) Inventors; and

(75) Inventors/Applicants *(for US only)*: HANSE, Eric [BE/BE]; 9, rue de la Gayolle, B–7370 Dour (BE). DUBOIS, Pascal [FR/FR]; 105, rue Pasteur, F–59750 Feignies (FR).

(74) Agent: DEBLED, Thierry; Vesuvius Group S.A., 17, rue de Douvrain, B–7011 Ghlin (BE).

(81) Designated States: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

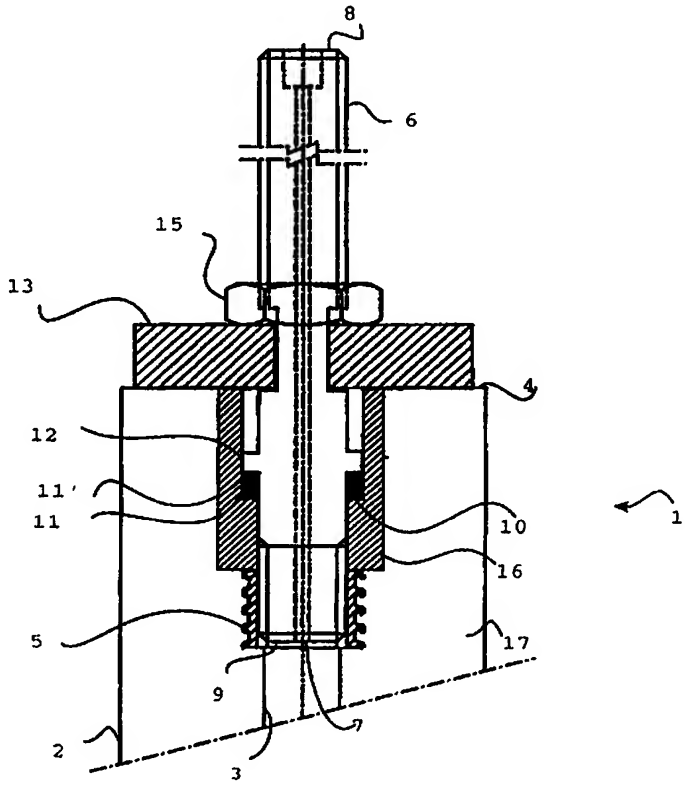
Published

With international search report.

(54) Title: STOPPER ROD

(57) Abstract

The present invention concerns a one–piece stopper rod whose gas tightness is improved. In particular, the stopper rod of the present invention has a body of refractory material comprised at least partially of a refractory material relatively impermeable to gases.



FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AL	Albania	ES	Spain	LS	Lesotho	SI	Slovenia
AM	Armenia	FI	Finland	LT	Lithuania	SK	Slovakia
AT	Austria	FR	France	LU	Luxembourg	SN	Senegal
AU	Australia	GA	Gabon	LV	Latvia	SZ	Swaziland
AZ	Azerbaijan	GB	United Kingdom	MC	Monaco	TD	Chad
BA	Bosnia and Herzegovina	GE	Georgia	MD	Republic of Moldova	TG	Togo
BB	Barbados	GH	Ghana	MG	Madagascar	TJ	Tajikistan
BE	Belgium	GN	Guinea	MK	The former Yugoslav Republic of Macedonia	TM	Turkmenistan
BF	Burkina Faso	GR	Greece			TR	Turkey
BG	Bulgaria	HU	Hungary	ML	Mali	TT	Trinidad and Tobago
BJ	Benin	IE	Ireland	MN	Mongolia	UA	Ukraine
BR	Brazil	IL	Israel	MR	Mauritania	UG	Uganda
BY	Belarus	IS	Iceland	MW	Malawi	US	United States of America
CA	Canada	IT	Italy	MX	Mexico	UZ	Uzbekistan
CF	Central African Republic	JP	Japan	NE	Niger	VN	Viet Nam
CG	Congo	KE	Kenya	NL	Netherlands	YU	Yugoslavia
CH	Switzerland	KG	Kyrgyzstan	NO	Norway	ZW	Zimbabwe
CI	Côte d'Ivoire	KP	Democratic People's Republic of Korea	NZ	New Zealand		
CM	Cameroon			PL	Poland		
CN	China	KR	Republic of Korea	PT	Portugal		
CU	Cuba	KZ	Kazakstan	RO	Romania		
CZ	Czech Republic	LC	Saint Lucia	RU	Russian Federation		
DE	Germany	LI	Liechtenstein	SD	Sudan		
DK	Denmark	LK	Sri Lanka	SE	Sweden		
EE	Estonia	LR	Liberia	SG	Singapore		

STOPPER ROD.

Description.

The present invention concerns a new stopper rod for regulating the flow of a molten metal from a pouring group, for example, steel or cast iron, from a distributor or a casting ladle, and more particularly a one-piece stopper rod that has means for attachment to a lifting mechanism. In a particular embodiment, the stopper rod also has means for introducing an inert gas, such as argon, into the molten metal bath during continuous casting operations.

Such a stopper rod and its use are well known to the skilled artisan, in particular by U.S. Patents 4,946,083 and 5,024,422 to which reference is made. Among others, these documents describe a one-piece stopper rod that can be attached to a lifting mechanism, comprised of:

a) an elongated body of refractory material that has a borehole positioned coaxially with respect to the body of the stopper rod and adapted to fixedly receive a metal rod for its attachment to a lifting mechanism. The axial borehole of the body of refractory material has an enlarged part with an annular sealing surface spaced away from the upper end of the body of refractory material. The means for attachment of the metal rod are generally positioned between the enlarged part and the lower end of the body of refractory material. At its lower end, the body of refractory material may have means for introducing gas into the molten metal bath; and

b) an elongated metal rod attached to the body of refractory material and having an axial borehole communicating in its lower part with the borehole of the body of refractory material. The rod has a collar carrying an annular sealing surface facing the annular sealing surface of the body of refractory material to create a gas tight seal. The upper end of the rod is adapted to be attached to a lifting mechanism that permits the vertical displacement of the stopper rod inside of a pouring group such as a distributor. Means for fastening to the body of refractory material are generally positioned between the collar and the lower end of the metal rod.

The stopper rod may be connected to a gas supply line, generally but not obligatorily, through the upper end of the rod.

When such a stopper rod is used, the gas introduced is conveyed to the axial borehole of the body of refractory material in its lower part. Thanks to the means for introducing gas into the molten metal bath, that the body of refractory material has in its lower part, the stopper rod permits the introduction of gas into the molten metal bath. The annular sealing surfaces of the rod and the body of refractory material facing each other prevent substantial losses of inert gas and the infiltration of air.

To improve this tightness even more, it was proposed to place an annular gas tight

gasket between these sealing surfaces. U.S. Patent 4,946,083, for example, indicates that when a gasket with a thickness of ca. 0.4 mm and of material resistant to high temperatures, e.g., graphite, is in place, the interface between the annular sealing surfaces of the rod and the body of refractory material furnishes a tightness capable of resisting a pressure up to 3 bars.

This seal is essential for casting high-grade molten metal. In the first place, it is necessary to assure a good protection against the infiltration of air responsible for oxidizing the molten metal during pouring. On the other hand, when an inert gas is injected through the stopper rod, it is also indispensable to minimize the losses of inert gas that cause production cost overruns that are far from negligible.

The system in use at the present time still do not however furnish a completely satisfactory solution for these two points of view.

In pursuing his research in this domain, the applicant discovered that these problems are due to the fact that, for different reasons (unscrewing of the rod, expansion of the rod, ...), a loss of tightness could occur at the sealing joint between the annular sealing surfaces of the rod and the body of refractory material facing each other.

The applicant then discovered that the tightness of the stopper rod could be improved by using a body of refractory material of a particular type.

According to the present invention, a stopper having a body of refractory material in one piece, constituted at least partially of a refractory material relatively impermeable to gases is used.

The present invention thus concerns a one-piece stopper rod that can be attached to a lifting mechanism, comprised of:

(a) an elongated body of refractory material, that has

(i) a borehole positioned coaxially with respect to the body of the stopper rod and adapted to receive in a fixed manner a metal rod for its attachment to a lifting mechanism, the axial borehole of the body of refractory material having enlarged part that presents an annular sealing surface spaced away from the upper end of the body of refractory material;

(ii) means for attaching the said metal rod; and

(b) an elongated metal rod attached to the body of refractory material adapted at its upper end to be attached to a lifting mechanism for vertically displacing the stopper rod inside of a pouring group; that is characterized in that the said body of refractory material is comprised at least partially of a refractory material relatively impermeable to gases.

According to a particular embodiment of the invention, the stopper rod can be connected to a gas supply line. Therefore, said elongated body of refractory material has, at its lower end, means for introducing gas into the molten metal bath and said metal rod has an axial borehole communicating in its lower part with the borehole of

the body of refractory material.

By material relatively impermeable to gas, is meant a material whose specific permeability (expressed in m^2) in the vicinity of the working temperature of the stopper rod is less than that of the materials conventionally used. Preferably, the specific permeability in the vicinity of the working temperature of the stopper rod of relatively impermeable refractory material is less than half that of the materials conventionally used. The conventional materials generally have a specific permeability between 5.10^{-17} and $5.10^{-16} m^2$. The relatively impermeable materials that are preferred according to the present invention have a specific permeability less than $5.10^{-17} m^2$.

Suitable refractory materials relatively impermeable to gases are chosen among mixtures in which additives capable of reducing the mean diameter of the pores have been incorporated. These additives are well known to the skilled in the art. For example, fluxing agents such as the alkalis (Na_2O, K_2O, CaO, B_2O_3, ...), silicas etc. can be incorporated into the composition of the refractory material. Metallic elements that form carbides when brought to high temperature can also be incorporated. It is also possible to define the mean diameter of the pores by implementing a powdered composition whose granulometry is chosen to reduce the mean pore diameter. Obviously, one or more of these methods can be combined. Preferably fluxing agents are incorporated in the composition of the refractory material.

The applicant has found that it was uneasy to obtain a refractory material presenting a good compromise between the properties of impermeability and resistance to corrosion by molten steel. Thus, according to a particular embodiment of the invention, the body of refractory material is comprised of at least two different refractory materials; the body of refractory material having a first part comprised of a mixture relatively impermeable to the gases that substantially surrounds the region in which the sealing gasket is located and a second part comprised of refractory material resistant to corrosion by the molten metals. According to this embodiment, the part of the body of refractory material in contact with the molten metal is preferably comprised essentially of a refractory material resistant to corrosion, while the part comprised of a mixture relatively impermeable to the gases substantially surrounding the region in which the sealing joint is located is not in contact with the molten metal.

In this case, material relatively impermeable to gas is understood to be a material whose specific permeability in the vicinity of the working temperature of the stopper rod is less than that of the material or materials resistant to corrosion. Preferably, the specific permeability in the vicinity of the working temperature of the stopper rod of relatively impermeable refractory material is less than half that of the corrosion-resistant material.

Conventional corrosion-resistant materials generally have a specific permeability between 5.10^{-17} and $5.10^{-16} m^2$. The relatively impermeable materials preferred

according to the present invention have a specific permeability less than 5.10^{-17} m^2. The body of refractory material according to the present invention can be prepared by any of the conventional techniques well known to the skilled artisan; in particular, this body of refractory material can be prepared by cold or hot pressing or even by isostatic pressing. For reasons of facilitation, in the case where the body of refractory material is comprised of several different refractory materials, it is preferable to press at least one of the parts previously, generally the part that is less accessible. As a general rule, it is thus preferable to press that part of the body of refractory material enveloping the region in which the sealing gasket is located beforehand.

The stopper rod involved in the present patent application is essentially similar to that described in the U.S. Patents 4,946,083 and 5,024,422, to which reference is made. As a variant, it is also possible to use a stopper rod that also has means for maintaining the compression of the sealing gasket in contact with the annular sealing surface of the body of refractory material as described in our Belgian patent application filed on the same day as the present patent application.

Figures 1 and 2 are fragmentary views in cross section of the upper end of a stopper rod according to these modes of implementing the invention.

On these Figures, the stopper rod 1 is comprised of an elongated body of refractory material 2 with an axial borehole 4 extending from its upper end 4 to its lower end (not shown). The body of refractory material is provided with means for introducing inert gas (not shown) into the metal bath.

The body of refractory material also has means 5 for attaching a metal rod 6. The metal rod 6 also has an axial borehole 7 that passes through it from its upper end 8 to its lower end 9. The upper end 8 is adapted to receive a connector (not shown) for supplying an inert gas. Furthermore, the upper end 8 of the rod is adapted to be attached to a lifting mechanism (not shown). A gas under pressure, such as argon, is introduced into the axial borehole 3 of the body of refractory material by means of the rod 6 and is conveyed to the metal bath through the lower end of the body of refractory material.

The body of refractory material 2 has an enlarged part 10 that forms a sealing surface. Two graphite gaskets (11 and 11') rest on this sealing surface and thus prevent the infiltration of air or losses of inert gas.

The body of refractory material 2 is comprised of two different refractory materials; the body of refractory material is comprised of a first part 16 composed of a mixture relatively impermeable to the gases substantially surrounding the region in which the sealing gaskets 11 (and 11') are located, and a second part 17 composed of a refractory material resistant to corrosion by the molten metals.

In Figure 1, the metal rod 6 has a collar 12 that carries an annular sealing surface facing the annular sealing surface 10 of the borehole of the body of refractory material

so as to create a seal against gases.

In Figure 2, the sleeve 14 is fitted on the rod 6 and maintains the gaskets 11 and 11' under compression. The upper part of the sleeve is blocked by a washer 13, it in turn being retained by a nut 15.

The washer 13 is preferably in contact with the upper end 4 of the body of refractory material 2 to give the assembly an increased rigidity.

The sleeve 14 is comprised of a material having a coefficient of thermal expansion greater than that of the metal rod 6 and a length sufficient so that, under the effect of the temperature to which the stopper rod is brought during pouring, it expands sufficiently toward the lower end of the metal rod to at least compensate for the expansion of the metal rod.

Preferably, the expansion of the sleeve compensates essentially precisely for the expansion of the metal rod.

As can be seen in Figure 2, the sleeve 14 can project at the upper end of the body of refractory material 2 if this is necessary to permit a sufficient length of the sleeve.

The sleeve 14 is fitted on the metal rod 6 and forms with it a free assembly, turning, sliding or just sliding. The upper end of the sleeve 14 just butts on blocking means 13 and 15 located fixedly on the metal rod 6 so that, under the effect of expansion, the sleeve 14 lengthens axially only in the direction opposite the said blocking means.

The material constituting the sleeve as well as its length are chosen as a function of the dimensions of the materials constituting the metal rod (generally machined from a steel bar with a coefficient of thermal expansion of the order of $12.5\mu m°C^{-1}$) and the body of refractory material (typically comprised of a refractory material obtained by isostatic pressing with a coefficient of thermal expansion of $3\text{-}6\mu m°C^{-1}$).

The material constituting the sleeve as well as its length are easily determined from the basic principles of thermal physics.

Starting with the values thus determined in the first approximation and which generally furnish excellent results, it is then possible to optimize the system by trial and error without any difficulty.

According to the invention, the sleeve is comprised of a material with a high coefficient of thermal expansion capable of resisting the elevated temperatures to which the stopper rod is subjected during pouring. For example, refractory materials with a high coefficient of thermal expansion such as fritted magnesia can be used. The preferred materials for this application are found among metals or metal alloys with a high coefficient of thermal expansion and having a high melting point.

According to a particular embodiment of the invention, the stopper rod also has means for preventing the metal rod from separating from the body of refractory material. Such means are described in the Belgian patent application No. 9800838 to which reference is made. Thus, if a metal insert having a threaded axial internal borehole

anchored in the body of refractory material is used as the means for attaching the rod to the body of refractory material, the rod will be prevented from becoming unscrewed from the insert by furnishing it with a pair of parallel plane surfaces at the point of emergence from the body of refractory material and by supporting on these plane surfaces an integral forked flange joined fixedly to the body of refractory material. This fixed joint can be accomplished by a pin inserted in a shaft effected through the forked flange and extending into the body of refractory material.

Claims.

1. Stopper rod, comprised of:

a) an elongated body of refractory material having

(i) a borehole positioned coaxially with respect to the body of the stopper rod and adapted to receive fixedly a metal rod for its attachment to a lifting mechanism, the axial borehole of the body of refractory material having an enlarged part that presents an annular sealing surface spaced away from the upper end of the body of refractory material; and

(ii) means for attaching the said metal rod; and

b) an elongated metal rod attached to the body of refractory material adapted at its upper end to be attached to a lifting mechanism for vertically displacing the stopper rod inside of a pouring group;

characterized in that said body of refractory material is comprised at least partially of a refractory material relatively impermeable to gases.

2. Stopper rod according to claim 1, **characterized in that** said metal rod has an axial borehole communicating in its lower part with the borehole of the body of refractory material.

3. Stopper rod according to claim 1 or 2, **characterized in that** said body of refractory material has, at its lower end, means for introducing gas into the molten metal bath.

4. Stopper rod according to any one of claims 1 to 3, **characterized in that** the material relatively impermeable to gases is chosen among refractory materials incorporating additives capable of reducing the mean diameter of the pores, such as fluxing agents and/or metallic elements that form carbides when they are brought to a high temperature.

5. Stopper rod according to claim 4, **characterized in that** the material relatively impermeable to gases is chosen among materials incorporating fluxing agents chosen among alkaline oxides or silicas.

6. Stopper rod according to any one of claims 1 to 3, **characterized in that** the material relatively impermeable to gases is prepared from a powdered composition whose granulometry is defined to obtain a material with a reduced specific permeability.

7. Stopper rod according to any one of claims 1 to 6, **characterized in that** the material relatively impermeable to gases has a specific permeability less than half that of conventional refractory material.

8. Stopper rod according to any one of claims 1 to 7, **characterized in that** the

material relatively impermeable to gases has a specific permeability less than $5.10^{-17}m^2$.

9. Stopper rod according to any one of claims 1 to 8, **characterized in that** the body of refractory material is comprised of at least two different refractory materials, a first part comprised of a mixture relatively impermeable to gases substantially surrounding the region in which the sealing gasket is located, and a second part comprised of a refractory material resistant to corrosion by molten metal.

10. Stopper rod according to any one of claims 1 to 9, **characterized in that** the tightness between the rod and the sealing surface of the body of refractory material is assured by a collar located on the rod.

11. Stopper rod according to any one of claims 1 to 10, **characterized in that** it also has means for maintaining the compression of the sealing gasket in contact with the annular sealing surface of the body of refractory material.

12. Stopper rod according to claim 11, **characterized in that** it has a sleeve fitted on the rod as a means for maintaining the compression of the sealing gasket.

Fig. 1

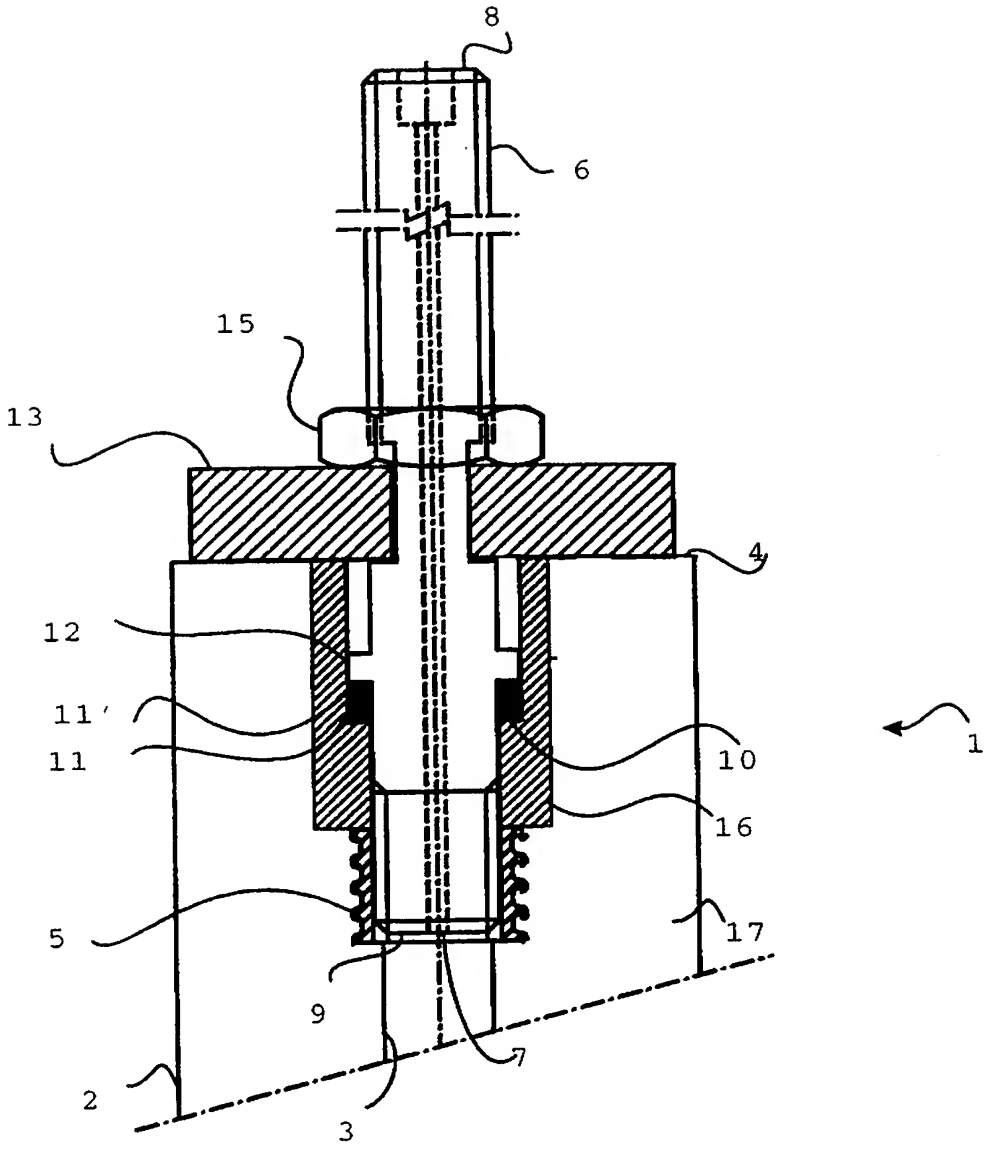
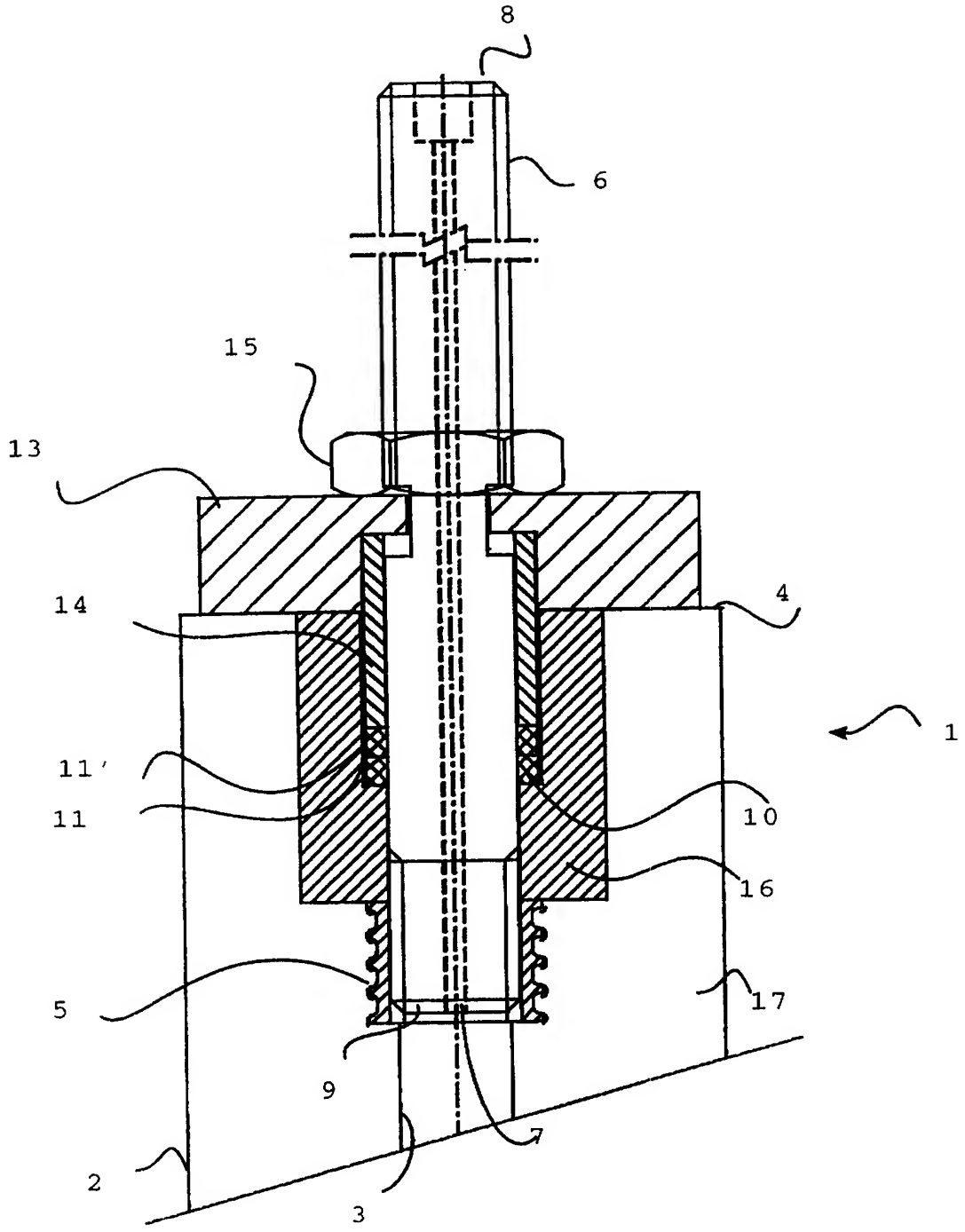


Fig. 2



INTERNATIONAL SEARCH REPORT

Interr ...nal Application No
PCT/BE 99/00146

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 B22D41/18

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 B22D C21C F27D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 4 791 978 A (FISHLER MARK K) 20 December 1988 (1988-12-20) column 3, line 13 - line 26 column 6, line 36 - line 46	1-3,6, 10-12
Y	US 4 946 083 A (FISHLER MARK K ET AL) 7 August 1990 (1990-08-07) cited in the application column 3, line 60 -column 6, line 24; figures 1-3	1-3,6, 10-12
A	US 5 024 422 A (FISHLER MARK K ET AL) 18 June 1991 (1991-06-18) cited in the application abstract; figure 2	1

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

° Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
3 February 2000	11/02/2000

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL – 2280 HV Rijswijk Tel. (+31–70) 340–2040, Tx. 31 651 epo nl, Fax: (+31–70) 340–3016	Mailliard, A

1

Form PCT/ISA/210 (second sheet) (July 1992)

INTERNATIONAL SEARCH REPORT

Information on patent family members

Intern nal Application No
PCT/BE 99/00146

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 4791978 A	20-12-1988	EP 0320481 A	14-06-1989
		JP 2006040 A	10-01-1990
US 4946083 A	07-08-1990	EP 0358535 A	14-03-1990
		JP 2182357 A	17-07-1990
		US 5024422 A	18-06-1991
US 5024422 A	18-06-1991	US 4946083 A	07-08-1990
		EP 0358535 A	14-03-1990
		JP 2182357 A	17-07-1990

Fig. 1

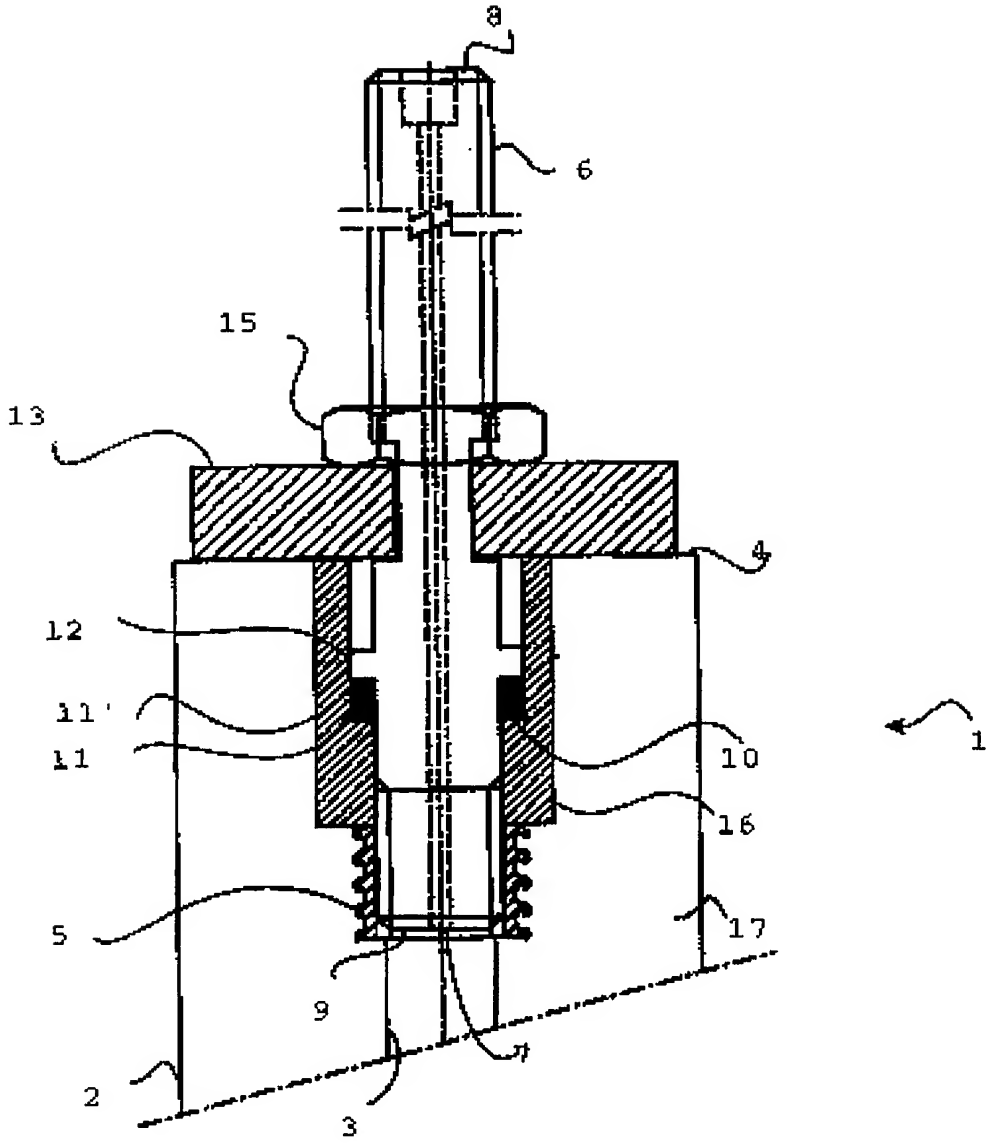


Fig. 2